

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.: 10/537,651
Applicant: Lindall *et al.*
Filed: October 31, 2005
Title: CATALYST AND PROCESS
T.C./A.U.: 1793
Examiner: McDonough, James E.
Confirmation No.: 5162
Docket No.: JMYS-128US

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION OF RICHARD WARD, Ph.D. PURSUANT TO 37 CFR §1.132

I, Richard Ward, Ph.D., being duly warned that willful false statements and the like are punishable by fine or imprisonment or both, under 18 U.S.C. §1001, and may jeopardize the validity of the patent application or any patent issuing thereon, state and declare as follows:

1. All statements herein made of my knowledge are true and statements made on information and belief are believed to be true.
2. I received a doctoral degree in Chemistry at the University of Durham in 2006. I received a masters degree in Chemistry from the same university, in 2003.
3. I have been employed by Johnson Matthey PLC since 2006 and have been with the VERTEC™ business unit since 2008. I am currently the Technical Development Manager and am responsible for the polymers research and development team, which includes ensuring the effective polyurethane catalyst product platform development and commercialization and running the technical support and product development for the polyester products. A copy of my *curriculum vitae* was attached with my last declaration.

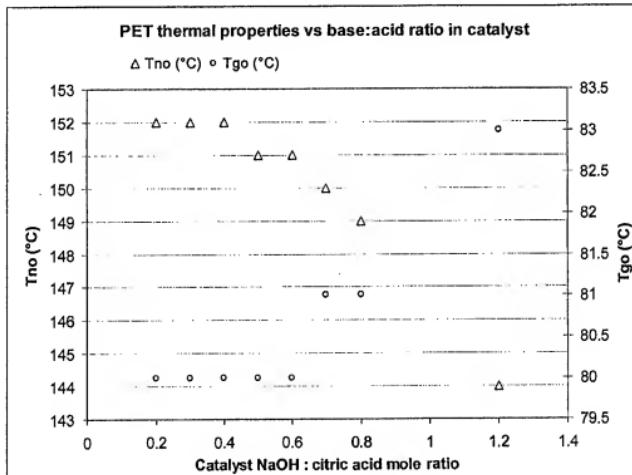
4. I have read, and am familiar with, the contents of this patent application, the Office Actions dated August 3, 2009 and January 13, 2010, and the cited reference, European Patent No. 0 812 818 (Ridland et al.). In regard to my previous declaration, the Examiner alleges that the prior declaration was not commensurate with the full scope of the claims and did not compare the closest prior art. This declaration addresses these issues.

5. The experiments discussed herein were conducted under my supervision.

6. Catalysts were prepared having mole ratios of base : acid of 0.2, 0.3, 0.4, 0.5, and 0.6, which are encompassed in the claimed range. Additionally, catalysts were prepared having mole ratios of base : acid of 0.7, 0.8, and 1.2, which are outside of the claimed range. The catalysts were all prepared according to Example 14 of the present application using an amount of sodium hydroxide calculated to provide the required base : acid mole ratio. A batch of polyethylene terephthalate (PET) was prepared using each of the catalyst compositions. A standard method of preparation was used which was the same for each batch, apart from the composition of the catalyst. Samples of the PET were investigated using DSC thermal analysis methods, following the procedure described on page 12, lines 19-24 of the present application. As described, the DSC measurement is carried out using a slow heating ramp to give an initial trace and then the chip is rapidly cooled. The chip is then subjected to a re-heating stage (same profile as first stage) and this produces a second trace, which is reported below:

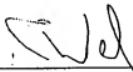
Catalyst NaOH:Citric Acid Ratio	Initial Heat					
	Tg _o (°C)	Tn _o (°C)	Tn (°C)	ΔH (J/g)	Tp (°C)	ΔH (J/g)
1.2	83	144	152	-20	251	28
0.8	81	149	159	-21	254	27
0.7	81	150	171	-25	255	32
0.6	80	151	161	-25	248	26
0.5	80	151	168	-21	254	24
0.4	80	152	165	-20	253	25
0.3	80	152	167	-14	252	25
0.2	80	152	170	-19	249	30

7. A plot of these results is provided below. In the plot below and in the Table in paragraph 6, T_{no} represents the temperature at which the onset of crystallisation is measured and is the point at which the crystallisation begins to take place and the polymer properties begin to change. T_n represents the crystallisation temperature. T_{go} represents the glass transition temperature.



8. As is evident from the plot, the thermal processing "window" between the glass transition temperature and the onset of crystallisation was found to be surprisingly wider when PET is made using the catalyst having a mole ratio of base : acid in the claimed range. The higher onset of crystallisation and crystallisation temperature (T_n) of PET provides a benefit when the PET is melt processed and oriented or drawn because the orientation can take place between a wider range of temperatures before the PET structure sets on crystallisation. Thus, polyesters made using the catalyst according to the invention, in the claimed base to acid range of 0.01 - 0.6:1, surprisingly exhibited a very low T_g (glass transition temperature) and a significantly higher onset of crystallisation temperature.

9. The raised crystallisation temperature in the PET made using the catalyst with a base to acid ratio in the range of 0.01 - 0.6:1 is unexpected and commercially important. This is because polyester resin is made into commercial articles, such as bottles, films, and fibers, by forming those articles from molten polyester. When the polyester has solidified from the melt, it is further processed by orienting the polymer molecules to form dimensionally stable and strong articles. This processing, which includes stretching bottles and films and drawing fibers, must take place at a temperature above the glass transition temperature (T_g) of the PET but below the crystallisation temperature of the PET because when the polymer is crystallised the oriented structure in the polymer is set. With the slower crystallisation exhibited by polyester made using the catalysts of the invention, the temperature "window" within which the PET processor can form and orient the polyester articles is significantly widened so allowing more processing to take place before the polymer crystallises. This is a significant benefit for a fabricator of polyester articles. This effect and the commercial benefit it imparts to polyester were wholly unexpected. I would not have anticipated being able to produce this effect from my knowledge of the background technology as described in the Ridland patent.



Richard Ward, Ph.D.

Date: 13/5/10.